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# A QUALITY ASSURANCE PROGRAM FOR THE EPA/SHAWNEE WET LIMESTONE SCRUBBER DEMONSTRATION PROGRAM

RESEARCH TRIANGLE INSTITUTE

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16. ABSTRACT The report describes a short-term quality assurance program, implemented at the EPA wet limestone scrubber facility located at the Shawnee steam/electric plant, Paducah, Kentucky. The program was part of a project to prepare a set of quality assurance quidelines for demonstration projects. The control laboratory, the effluent gas streams, and process instrumentation were reviewed and audited. In the control laboratory, side-by-side pH measurements were made, and limestone slurry samples were collected. These samples were sent to three independent laboratories for analysis of selected elements in the solid and liquid phases. Gas stream work covered both particulate grain loading and analysis of SO2. Particulate sampling and weighing techniques were observed, and volume calibration checks were made. SO2 was collected, analyzed by two chemical methods, and compared with the in-stack photometric measurement system. Process instrumentation was checked with portable precision electronic equipment carried on-site and inserted into instrumentation circuitry to verify accuracy of sensors and readout devices.

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## 1.0 INTRODUCTION

General aspects of demonstration program quality assurance (QA) programs have been treated in the report, "Guidelines for Demonstration Project Quality Assurance Programs."* The present report contains results of an external audit program carried out by the Research Triangle Institute** during the week of November 17-21, 1975, at the Shawnee project. The cooperation of TVA, EPA, and Bechtel Corporation personnel is acknowledged and gratitude for such cooperation is hereby expressed.

The approach taken here is similar to that used in the Interim Report (Subtask 2) for this project; namely, the program is arbitrarily divided into three major areas. These are the control laboratory, gas stream sampling and process instrumentation. Sections 2.0, 3.0 and 4.0 treat these areas, in the order mentioned. Section 5.0 presents recommendations for an external quality assurance program at the Shawnee Scrubber facility.

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*The guidelines report was prepared just prior to planning and implementation of the short-term quality assurance program at the Shawnee project.

**Research Triangle Park, N.C.

## 2.0 THE CONTROL LABORATORY

### 2.1 Measurement of pH

A major element for process control of the scrubber is pH measurement. For this reason a significant part of the RTI effort was spent in observing and verifying the TVA techniques for pH determination at the scrubber inlet and outlet. For audit purposes, a Fisher Research Grade pH Meter (Acumet Model 320 with expanded scale) was equipped with a Markson #1888 Polymark Combination pH electrode fitted with a 20-foot lead. The long lead allowed measurement in various slurry "pots" on the scrubber without moving the meter. TVA technicians routinely use the same type probe, but on a less sensitive meter. In spite of the claimed ruggedness of the probe, it was said by TVA personnel that some thirty had been purchased, used and discarded over a period of a year. The probes are necessarily subjected to much physical abuse.

A portable pH system is used several times a day by TVA operators to check the control room readings obtained from Universal Interloc, Inc., Model 321 Submersion pH sensors. These devices are permanently mounted in pots through which slurry continuously circulates when the scrubber is operating. Four such sensors monitor the inlet and outlet pH for the TCA* and Venturi scrubbers. The system for checking and recording pH is as follows: at least six times a day (twice each 8-hour shift) an operator goes out to the scrubber for sample collection and pH reading. A small metal shed on the scrubber holds the pH meter, buffer solutions, and associated gear. The operator calibrates the meter by means of a buffer solution of known pH. The buffer pH is selected so as to be within one unit or less of the actual slurry pH. The temperatures of both the buffer and the slurry are checked, and the temperature compensating potentiometer on the meter is adjusted accordingly.** A study by Bechtel personnel, underway at the time of the RTI audit and independently verified

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* Turbulent Contact Absorber.

** It was observed that these temperatures were not regularly measured, sometimes being estimated. This is not likely a significant error source, since the pH should be relatively insensitive to a temperature variance of a few degrees centigrade.

by RTI personnel, showed that the combination probe took a finite time to respond to solution temperature differences. Over the range from 20° to 50° C (typical for buffer-slurry temperature differences) the response time was estimated at 4 to 5 minutes for heating and 10 to 15 minutes for cooling. A constant temperature bath operated by Bechtel was also used by RTI in making this study. As a result of this study, a directive was issued by TVA that operators wait at least 5 minutes before recording slurry pH, to allow for probe warmup after standardization with ambient temperature buffer. Also, rapid switching from slurry to buffer or buffer to slurry was forbidden. This may well improve the accuracy of the technique, since over a 30-degree range the pH error is about 0.1 unit, which is significantly large.

After recording the indicated slurry pH, the operator uses a paging telephone to call down to the control room. Technically, the operator is not allowed to know the pH being read by the inline probe. A control room operator records the pH called down and compares it with the inline probe measurement. The inline probe reading is adjusted if the difference is considered too large. The portable pH system is used as a standardization system for the permanently mounted probes.

In practice it was found that the reverse was sometimes true; i.e., the portable system was adjusted to deliver the "expected" pH of the Uni-Loc probe. This was the case particularly at certain test points where the Markson probe was inserted in a horizontal pipe fitted into the side of the slurry outlet pot. Measurement of pH by means of these pipes (equipped with a faucet valve) has now been discontinued, so a detailed discussion of the problems associated with such measurements is unwarranted.

A series of direct comparison pH measurements were made on November 19-20, 1975. RTI personnel set up a Fisher Acumet meter beside the TVA (Orion) meter and made simultaneous measurements of slurry pH. The operators were instructed to carry out their measurements routinely, from standardization to cleanup. Also during this period the Uni-Loc probes were removed from their pots and immersed in pH 5 and 6 buffers. Both RTI and TVA long-lead probes were put into these same buffers, after independent standardization (RTI standardization was against Fisher Certified Buffer Solution). The values obtained in the buffer and in slurry are summarized in table 1. Values of pH were read to the

Table 1. Comparisons* for pH

| TEST POINT | pH RTI (portable) | pH TVA (portable) | pH TVA (inline) | Temperature (°Centrigrade) | Date (mo/day/yr) | Time (hours) |
|---|---|---|---|---|---|---|
| 1816[1] | 5.25 | 5.3 | 5.16 | 54 | 11/19/75 | 11:15 |
| | 5.13 | 5.2 | 5.29 | 54 | 11/19/75 | 15:30 |
| | 5.05[5]** | 4.9 | 5.04 | 16 | 11/20/75 | 08:45 |
| | 5.24 | 5.2 | 5.34 | 54 | 11/20/75 | 09:10 |
| | 5.28 | 5.2 | 5.38 | 50 | 11/20/75 | 09:45 |
| | 5.34 | 5.3 | 5.34 | 51 | 11/20/75 | 11:15 |
| | 5.06 | 5.1 | 5.25 | 50 | 11/20/75 | 15:15 |
| 1825[2] | 4.92 | 4.9 | 5.17 | 53 | 11/19/75 | 11:30 |
| | 4.83 | 4.9 | 5.02 | 54 | 11/19/75 | 15:30 |
| | 4.77 | 4.8 | 5.08 | 50 | 11/20/75 | 15:15 |
| 2816[3] | 5.03[5] | — | 4.82 | 21 | 11/19/75 | 10:00 |
| | 6.02[6] | 6.0 | 5.77 | 21 | 11/19/75 | 10:30 |
| 2825[4] | 5.03[5] | 5.0 | 4.99 | 21 | 11/19/75 | 10:50 |
| | 6.01[6] | 6.0 | 6.12 | 21 | 11/19/75 | 11:00 |

1 Venturi effluent hold tank.
2 Venturi outlet.
3 TCA effluent hold tank.
4 TCA outlet.

*Unless otherwise noted, measurements are on slurry in inline probe pots.

**Superscript number indicates measurement of a buffer solution of pH 5 or 6.

nearest 0.01 unit using the Acumet expanded scale, but readings of such precision were not possible with the Orion meter, where estimations to 0.1 pH unit were made. Actual markings on the Orion meter scale were at 0.2 pH unit intervals.

Two observations are in order, after study of table 1:

1) The RTI and TVA portable (long-lead) pH systems agreed within 0.1 pH unit or better in every comparison made. This verifies the accuracy of the TVA portable system, since the RTI system was standardized against certified buffer. Operator reading errors are probably the largest error source in the pH measurement process.

2) The Uni-Loc system readings differed from RTI readings by 0.0 to 0.3 pH unit, with the mean difference being 0.14 unit over 14 readings. Certainly it would be unwise to dwell on the significance of the statistics of such a brief study. One point can be made, however, with respect to the confidence placed in the Uni-Loc pH readings. It appears unlikely that, using the present system of inline probes, pH measurements on the slurry can be made to better than 0.1 unit. There are a number of factors which militate against greater accuracy, the major one probably being the nature of the slurry itself. This viscous, highly abrasive suspension tends to clog lines and coat out on probe surfaces, thus making reproducible measurements quite difficult. The non-equilibrium mixture of reactive chemicals has a pH which will change on removal from the scrubber proper; i.e., as it flows into the pots within which measurements are made. Another factor is the difficulty of standardization of inline probes. The present system calls for probe removal, cleaning and standardization roughly each 2 days. The accuracy of the pH measurement is surely dependent on the condition of the probe surface, and restandardizing is ideally done shortly before each measurement.

## 2.2 Analysis of Slurry

As a check on the reliability of the chemical analysis phase of the scrubber operation, a series of slurry samples was collected* and sent to

*All samples were taken from the venturi effluent hold tank.

several other laboratories for independent analysis of both the liquid phase and suspended solids.

The laboratories originally selected for participation in this phase of the audit were two TVA laboratories (Chattanooga and Muscle Shoals), EMSL (EPA-RTP) and RTI. The EMSL laboratory later declined to participate in the project.

Each laboratory was given five 1-liter samples of slurry, taken concurrently with control laboratory samples. After filtering and drying the solid, it was analyzed for calcium, magnesium, and total sulfur. The filtrate was analyzed for calcium, magnesium, solium, potassium, and chloride. As a check on filtration technique the RTI laboratory was given, in addition to samples of slurry, samples which were filtered in the control laboratory. The filtrate and dried solid were then analyzed in the same way as the unfiltered slurry samples.

Complete results of these analyses are given in appendix A. Results for one sample, collected at 3 p.m. on November 18, are summarized in table 2.

No statistical analyses have been performed on these data, in view of the short time period for data collection and the small number of samples analyzed. The data serves merely as representative of the type obtainable by means of split samples. One observation is pertinent at this time; namely, that each cooperating laboratory was strikingly consistent in its analysis of each element, with large average differences in between-laboratory results. Part of this can be attributed to use of different analytical techniques by different laboratories. In an ongoing program involving several laboratories it would be necessary to evaluate each analytical technique as to bias. The comprehensive final report for this project will attempt to evaluate each individual technique used, for comparative purposes.

Liquid analysis at the control laboratory was as follows: calcium, magnesium, sodium, and potassium were done by atomic absorption (AA) and chloride by potentiometric titration. Caldium, magnesium, and total sulfur analysis in the solid were done by X-ray fluorescence (XRF). The XRF standard was a sample of slurry solids sent out to three other TVA laboratories by Mr. Barkley, chief chemist at the control laboratory. Results of the independent laboratory analyses were averaged and assigned as standard values for calcium (as CaO),

Table 2. Analysis of slurry sample

| Element | Shawnee control laboratory | | | RTI (Shawnee filtered) | | RTI (RTI filtered) | | Chattanooga | Muscle shoals |
|---|---|---|---|---|---|---|---|---|---|
| Liquid (in ppm) | | | | | | | | | |
| Ca | 1710 | | | 1825 | | 1810 | | 1731 | 1787 |
| Mg | 699 | | | 945 | | 805 | | 742 | 724 |
| Na | 57 | | | 69 | | 161 | | 62 | 37 |
| K | 121 | | | 101 | | 102 | | 96 | 54 |
| Cl | 3580 | | | 3700 | | 3638 | | 3543 | 3700 |
| Solid | | | | XRF | | | XRF | | |
| (in wt %) | | AA[1] | SS[2] | RTI[3] | AA | SS | RTI | | |
| CaO | 24.50 | 19.01 | 25.41 | 22.06 | 17.96 | 25.52 | 22.16 | 23.24 | 23.1 |
| MgO | 0.29 | 0.48 | | | 0.52 | | | 0.56 | 0.25 |
| TS($SO_3$) | 34.16 | 32.00 | | 21.22 | 26.96 | | 30.14 | 30.7 | 30.9 |

[1]Atomic absorption, on acid-dissolved solid.
[2]Using values given by Shawnee control laboratory for XRF standard.
[3]Using values obtained by RTI for Shawnee XRF standard.

Table 3. Analysis of XRF standard

| Element | Shawnee value (wt %) | RTI value (wt %) |
|---|---|---|
| Ca | 18.15 | 15.76[1] |
| Mg | 0.185 | 0.275[1] |
| S | 11.38 | 11.35[2] |

[1]Analyzed by atomic absorption after acid dissolution and dilution.
[2]From precipitation with barium chloride.

magnesium (as MgO) and total sulfur (as $SO_3$). A portion of this standard was taken for analysis by RTI and for use as the RTI XRF standard. Analysis results for the standard are presented in table 3.

Use of the same XRF standard by both Shawnee and RTI precluded a discrepancy in results due to a matrix effect. Agreement was excellent for sulfur, fair for calcium, but poor for magnesium. Because of differences in standard assignment values for calcium, two XRF results are given, as explained in the footnote to table 2. Magnesium could not be determined with the XRF instrumentation used by RTI.

## 2.3 Overall Laboratory Evaluation

In summation, the control laboratory operation appears to be adequate for the routine analytical work it performs. It has no formal Quality Control program, but bad data may be flagged by either TVA or Bechtel personnel. Acceptance limits on data are not formalized, but "reasonableness" is the experience-based criterion.

There are problems associated with the lack of operator training programs, incentives for superior performance and the like, but so long as the laboratory operations remain strictly routine these problems are not likely to seriously hamper the program.

Equipment and instrumentation are approprite for the type of work done, and it is maintained on a regular basis (largely by service contracts).

Results of the qualitative systems review for the laboratory are included as appendix B of this report.

## 3.0 EFFLUENT GAS STREAM SAMPLING

### 3.1 Particulate Mass Loading

Side-by-side duplicate runs were not attempted. The entire sampling procedure was observed, with critical techniques observed repeatedly, during the site visits. Overall performance was evaluated using the checklist given as table 4. On a scale of 1 to 5, ranging from unacceptable to excellent, the Shawnee particulate loading technique was rated 3 (acceptable). Specific comments are given below.

#### 3.1.1 Pitot Tube Comparison

A comparison of TVA and RTI pitot tubes was performed at the Venturi inlet only. A check of the outlet tube was not carried out due to the outlet tube misalignment ($\geq 30°$) along its roll axis.

Side-by-side measurements were performed. Based upon comparison with the RTI (NBS calibrated) pitot tube, the TVA tube Cp factor was 0.879. The assumed value was 0.850. The difference was considered to be negligible.

#### 3.1.2 Temperature Measurement

A system capable of measuring the stack gas temperature to within 1.5 percent of the minimum absolute stack temperature is required. The temperature-measuring system (inlet sampler) was checked versus a calibrated thermocouple and was found to be within 1 percent.

#### 3.1.3 Moisture Measurement

The impinger section of the EPA sampling train is intended to collect moisture from the sample gases for determination of moisture content. The last impinger contains silica gel to adsorb the water vapor not condensed in the first two impingers. The moisture content of the sample gas leaving the silica gel impinger increases as the exit gas temperature rises. Also the exit gas moisture content will increase as the sample train vacuum increases at any one sample temperature. Moisture not collected by the condensation system is incorrectly measured as dry gas by the dry test meter and the error

Table 4. Particulate emission evaluation checklist

| YES | NO | OPERATION |
|---|---|---|
| | | EQUIPMENT PREPARATION AND CHECK |
| ___ | ___*1 | 1. Sampling train assembled and leak-checked. |
| ✓ | ___ | 2. Probe and filter box heaters checked and set for proper temperatures. |
| ___ | ✓ | 3. Stack gas temperature measuring system assembled and checked for proper operation by comparing to a mercury in glass thermometer. |
| ✓ | ___ | 4. Stack gas velocity measuring system assembled and checked for proper operation. |
| | | PRELIMINARY MEASUREMENTS |
| ✓ | ___ | 5. Selection of traverse points according to Method 1. |
| ___ | ✓ | 6. Moisture content by Method 4, or equivalent. |
| ✓ | ___ | 7. Molecular weight by Method 3, or equivalent. |
| ✓ | ___ | 8. Measurement of stack dimensions. |
| ✓ | ___ | 9. Mark probe for sampling at traverse points. |
| | | SAMPLE COLLECTION |
| ✓ | ___ | 10. Equal sampling time at each traverse point |
| ___ | ___*2 | 11. Probe temperature satisfactory throughout the test. |
| ✓ | ___ | 12. Filter box temperature 120° C ± 15° (250° F ± 25°) through the test. |
| ___ | ___*2 | 13. Sample gas temperature at last impinger ∿ 21° C (70° F) throughout the test. |
| ✓ | ___ | 14. Isokinetic sampling checked and adjusted if necessary at least every 5 minutes. |
| ___ | ✓ | 15. Leak-check |
| | | SAMPLE RECOVERY |
| ___ | ✓ | 16. Satisfactory handling and movement of probe and filter to sample recovery area. |

*1 Not observed.
*2 Not monitored.

Table 4. Particulate emission evaluation checklist (continued)

| YES | NO | OPERATION |
|---|---|---|
| ✓ | | 17. Recovery area satisfactory (i.e., space, cleanliness, etc.). |
| | ✓ | 18. Sample recovery procedure adequate. |
| ✓ | | 19. Proper labeling of sample containers. |
| | ✓ | 20. Determination of moisture content procedure adequate. |
| | | ANALYSIS |
| | *3 | 21. Proper equilibration of (1) filter, (2) probe wash residue, and (3) acetone blank residue. |
| | *3 | 22. Correct collected particulates for acetone blank. |
| | *1 | 23. Analytical balance checked before weighings. |
| | | DOCUMENTATION |
| ✓ | | 24. All information recorded on data sheet as obtained. |
| | | 25. All unusual conditions recorded. |
| | | COMMENTS |

*3 Probe wash and acetone blank residue not measured.
*1 Not observed.

is carried through the isokinetic and grain loading calculations. However, if the exit gas temperature is held below 25° C and the rain vacuum is held below 380 mm of Hg, the resulting error in the sample volume will be less than 2 percent. A single RTI reading of exit gas temperature was 22° C.

There was evidence of significant moisture accumulation in the silica gel, indicating the presence of some water vapor in the total gas volume measured. This does not likely introduce a large error into the technique, although it would be advisable to make quantitative or semiquantitative checks on the actual water volume collected versus water content of the stack gas. This is not presently being done at Shawnee.

### 3.1.4 Volume Measurement

The sampling train was checked for accuracy of volumetric measurement with a calibrated dry test meter (1 cf/revolution) which had been previously calibrated versus a 1 cf wet test meter. The RTI meter was connected directly to the Shawnee probe tip, so that the actual volume intake at the probe was measured. RTI volume was 15.8 percent lower than TVA volume, indicating a rather large positive bias in the TVA measurement. Critical examination of the TVA sampling system led to the conclusion that the bias could be attributed to leaks in the system (broken or cracked polycarbonate impinger tubes, loose probe tip, etc.). Leakage rate was estimated to be 0.36 cfm at 380 mm of Hg vacuum.* Inaccuracies in volume measurements appear directly in the concentration and particulate mass emission rate determinations.

A probe tip diameter check was made with a micrometer. The range of the diameter measurements was 0.7 mm, indicating a severely out-of-round nozzle which should be repaired or replaced. The estimated nozzle area was calculated to be roughly 20 percent lower than the assumed area (0.583 $cm^2$ calculated, 0.7125 $cm^2$ assumed). An error in the nozzle diameter is quadrupled in the process of determining isokinetic sampling rates and is doubled in the percent of isokinetic sampling calculation. The percent isokinetic, as calculated with

*As indicated in table 4, TVA leak-checking was not observed by the audit team. A thorough leak-check would surely have detected such a significant leak-rate.

respect to the above errors in volume measurement and nozzle diameter could result in either a positive or negative bias, depending upon which factor predominates.

### 3.2 Sulfur Dioxide Concentration Determinations

Sulfur dioxide concentrations at the wet limestones scrubber facility are determined by means of du Pont Model 400 Photometric Analyzers. The analyzers continuously monitor inlet and outlet gas streams of the venturi and TCA units. They constitute a critically important measurement system, and as such a concentrated effort was made to assess their performance.

The RTI audit team collected a total of 23 gas samples, all collected at the venturi inlet. Thirteen of these samples were analyzed by the barium chloranilate (colorimetric) method, the remaining ten by sodium hydroxide titration. Results are given in table 5. The average bias of the photometric method with respect to the wet chemical methods was +6.9 percent, with a standard deviation of 8.7 percent. These results indicate that the du Pont analyzer at the venturi inlet is yielding data of high validity. At the 95-percent confidence level, an individual photometric determination should be within $\pm$18 percent of the true $SO_2$ mean concentration, biased 7 percent high on the average, based on the audit data. Due to the time limitation of the audit team it was not possible to run checks on the other three analyzers. Details of the RTI analytical procedures are given in appendix C.

Table 5. Comparison of $SO_x$ determinations

| Date | Sample Number | Sample Time | Sampling Train No.1: $SO_2$ by Barium Chloranilate (ppm) | Sampling Train No.1: $SO_x$ Contained in Isopropranol Scrubber (ppm) | Sampling Train No.1: Total | Sampling Train No.2: $SO_x$ by NaOH Titration* | Sampling Train No.2: $SO_2$ by TVA (DuPont Analyzer) | Sampling Train No.2: $\frac{TVA-RTI}{RTI} \times 100$ |
|---|---|---|---|---|---|---|---|---|
| 11/18/75 | 1 | 17:15-17:25 | | | | 2562 | 2750 | +7.3 |
| 11/18/75 | 2 | 17:35-17:45 | | | | 2252 | 2750 | +9.0 |
| 11/19/75 | 3 | 10:15-10:23 | 4187 | 543 | 4730 | | 3625 | - 23.3 |
| 11/19/75 | 4 | 10:42-10:50 | 2736 | 650 | 3386 | | 3585 | +5.9 |
| 11/19/75 | 5 | 11:07-11:21 | Sample Voided | --- | ---- | | 3366 | --- |
| 11/19/75 | 6 | 12:08-12:17 | Sample Voided | --- | ---- | | 3167 | --- |
| 11/19/75 | 7 | 12:43-12:49 | | | | 3053 | 3051 | -0.1 |
| 11/19/75 | 8 | 12:53-13:02 | 2261 | 530 | 2791 | | 3046 | +9.1 |
| 11/19/75 | 9 | 13:39-13:47 | | | | 2832 | 3016 | +6.5 |
| 11/19/75 | 10 | 13:50-13:59 | 2172 | 563 | 2735 | | 2985 | +9.1 |
| 11/19/75 | 11 | 14:22-14:31 | | | | 2812 | 2998 | +6.6 |
| 11/19/75 | 12 | 14:32-14:41 | 2309 | 530 | 2839 | | 2947 | +3.8 |
| 11/19/75 | 13 | 15:06-15:15 | | | | 2757 | 2893 | +4.9 |
| 11/19/75 | 14 | 15:17-15:27 | 1958 | 551 | 2509 | | 2909 | + 15.9 |
| 11/20/75 | 15 | 09:16-09:27 | 1582 | 523 | 2105 | | 2506 | + 19.0 |
| 11/20/75 | 16 | 09:30-09:40 | | | | 2435 | 2509 | +3.0 |
| 11/20/75 | 17 | 09:53-10:04 | 1827 | 643 | 2470 | | 2505 | +1.4 |
| 11/20/75 | 18 | 10:05-10:15 | | | | 2385 | 2508 | +5.2 |
| 11/20/75 | 19 | 10:22-10:33 | 1750 | 610 | 2360 | | 2570 | +8.9 |
| 11/20/75 | 20 | 10:41-10:52 | | | | 2459 | 2600 | +5.7 |
| 11/20/75 | 21 | 10:58-11:09 | 1724 | 541 | 2265 | | 2606 | + 15.1 |
| 11/20/75 | 22 | 11:11-11:22 | | | | 2294 | 2648 | + 15.4 |
| 11/20/75 | 23 | 11:37-11:48 | 1690 | 637 | 2327 | | 2719 | + 16.8 |

* $SO_2$ analyzer accepted on basis of a total acid determination (TVA analysis)

Average Bias: +6.9%

Std. Dev.: 8.69%

95% Confidence Interval $\pm$ 18.08%

## 4.0 PROCESS INSTRUMENTATION

In a continuing effort to establish guidelines for a comprehensive quality assurance program to be implemented at the EPA/TVA Shawnee Scrubber demonstration project and to further the overall QA program for demonstration projects, a set of quantitative data was scheduled to be taken* onsite by monitoring actual instrumentation working conditions. A series of calibration tests were planned using in-house calibrating equipment and an RTI precision digital multimeter (DMM) as a voltage and current monitor.

Three types of sensors (temperature, differential pressure, and flow rate) are the primary sources of measurement information being recorded and used for the scrubber's mechanical operation control. Each sensor's output is translated by its associated transmitter into a direct current, which passes through the series-connected set of control room monitoring instruments. This scheme has the advantage of having the same information (current value) applied to all readout devices alike, making wiring from the transmitter less critical than in a potential system (which requires parallel connections for the readout devices).

Four readout devices are employed for visual display of the sensor's output signal. Three of the devices, the Data Acquisition System (DAS), the Foxboro Trend Recorders, and Foxboro Process Controllers convert the transmitted current to a potential which is developed across a precision input resistance. The resistance is an integral part of the transmitter's current loop. The potential is electronically amplified for further processing. The stability of the resistance values of these input resistors is critical to the accuracy of measurement. The fourth device, a Foxboro strip chart recorder, uses a galvanometer movement (pen motor) to directly convert current into a proportional movement of the recorder pen. Both the strip chart units and the trend recorder units produce a continuous real-time record of the transmitted current value.

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*Daily work schedules of TVA process personnel would not allow completion of the entire testing program over the short on-site time period. Enough was accomplished for a judgment to be made as to the quality of the instrumentation facilities.

The methods of test and calibration are simple, using rudimentary sources of stimuli for sensor examination. Straightforward electrical current measuring instruments are used to monitor currents produced by the transmitters. The tests as performed are sufficient to maintain the quality of measurement to the degree established by the manufacturers in their design specifications. Calibration of each measurement channel (consisting of a sensor, a transmitter, and one or more readout devices) is performed by the instrument technicians. First, the sensor is subjected to a known stimulus under TVA instrument shop conditions (with essentially no temperature or humidity controls). The transmitter current level is monitored with a Foxboro Test Set/Calibrator Model 8121. Second, sensor and transmitter are connected in their normal operation positions and the DAS readout is used to monitor the transmitter's current level while stimulating the sensor with a known signal. One important part of any calibration procedure that is not presently being done at Shawnee is the recording of data so that a calibration curve can be generated. The calibration curve is useful in checking the linearity of the system as well as out-of-tolerance points between zero and full scale. Calibration curves made at a later time can be monitored for changes in instrument performance that could signal an impending instrument failure. For flow measurements a Foxboro Model 8120 Magnetic Flow Calibration is used in place of the sensor for preliminary transmitter/indicator calibration. Final flow measurements are calibrated by pumping known quantities of water for a measured time interval through the sensor and comparing the DAS reading with the water measurements. The above calibration measurements are performed using methods described in the Foxboro instruction manuals. Methods found necessary for setup and calibration of sensors, transmitters, and indicators for inline operation have not been written and referenced. The lack of written instructions does not necessarily indicate that the calibration work done at the Shawnee scrubber has been inferior. The crux of the matter, as far as a quality assurance program is concerned, is the complete lack of records pertaining to what has been done, when it was done and how accurately.

Due to the TVA work schedule only one laboratory calibration procedure was observed in the maintenance shop. Calibration of a Foxboro liquid level sensor and transmitter Model 617FM was accomplished using the Foxboro Model

8121 calibrator and a mercury-filled manometer. The sensor was subjected to an air pressure monitored by the manometer. The air pressure was varied from 0 inches Hg to 16.5 inches Hg, representing zero and full scale values respectively. The table below lists the current output for measured values of air pressure being applied. No recordings of instrument performance prior to readjustment were available. The mercury manometer used in the above calibration was readable to 0.1 inch of Hg, which is not sufficient for calibrating instruments requiring less than 10 inches of Hg pressure for full-scale operation.

The Fluke DMM was calibrated at an authorized factory service center and certified to be within the 0.005 percent accuracy specifications for that model instrument. The 100-ohm resistor was verified at the above facility to be 100.000 ohms $\pm$0.005 percent. Based on the above data, the Foxboro liquid level sensor and transmitter were calibrated to an accuracy exceeding the $\pm$1.0 percent value specified by the Foxboro Company. A significant part of the instrument's calibration is the ability of the readout device to indicate the proper numbers. The Data Acquisition System digital readout is capable of a $\pm$0.1 percent (of full scale) accuracy. Twelve of the 25-ohm precision resistors used by the DAS to monitor loop current were measured by the Fluke DMM. All were within 0.2 percent of the nominal value. Continuous operation of the DAS prevented an accurate check of the system. It was noted, however,

Table 6. Calibration of liquid level sensor

| Manometer reading (inches Hg) | Foxboro indicator current (milliamps) | Fluke DMM reading across 100.0 ohms (volts) |
|---|---|---|
| 0 | 10.0 | 1.001 |
| 4.1 | 20.01 | 2.002 |
| 8.2 | 30.02 | 3.004 |
| 12.3 | 40.01 | 4.003 |
| 16.5 | 50.00 | 5.001 |

that no data was recorded by the technician (in the form of a table) during his calibration procedure. The only record made was a note on the instrument's record card that the instrument had been checked and found to be satisfactory for service. Such records are not proof of the instrument's ability to perform within specifications. Since no previous calibrations performed on this instrument were recorded, it was impossible to determine what general behavior the instrument had followed. No trends of performance fluctuations could be derived. This information is vital to a good quality assurance program. Records are valuable in determining the nominal rate at which instruments must be removed from service, repaired, and recalibrated in order to keep their performance within tolerance requirements. The above test also demonstrated the ability of the Foxboro Model 8121 Calibrator to measure the transmitter current to an accuracy of better than $\pm$1.0 percent. There were no records available for this calibrator which would indicate the instrument's accuracy or shift in calibration from month to month. Since this instrument is used in the scrubber environment, making it subject to much abuse, it should be calibrated against a laboratory standard at least once a month. The same procedure should be followed for all other instruments used as general purpose calibrators. Calibration procedures for these instruments need to be developed and performed by one technician who is qualified for the work. That technician should be the only person having access to the laboratory standard instruments used to calibrate all other shop instruments, such as the Foxboro Model 8121 Calibrator. The laboratory standard instruments should be confined to a specific area in the shop and kept secure from use by other personnel.

The performance of equipment can only be judged by the review of accurate records which clearly show a life history of each item having a functional part in the operation of a system. An individual should be made responsible for keeping all records in a restricted area. Another function of record-keeping is to maintain up-to-date instruction manuals for each instrument being used. This would include the manufacturer's manuals, in-house use manuals, and calibration instructions as well as a history of performance data. An up-to-date log of the disposition and condition of each instrument on inventory should be kept, for the determination of overall accuracy of data describing the system's performance. This log would keep management informed

about the availability of spares for any changes or modifications that become necessary. It would also be useful to personnel in the instrumentation group for making necessary changes in equipment, should their supervisor not be available during an emergency.

A significant part of any quality assurance program is a formal audit of the work being performed by a qualified auditor. There was no evidence to show that an auditing program exists at the scrubber, nor has any attempt been made to have competent personnel make checks of the work being done by operating personnel. An audit program requires careful study and analysis of records being kept, and over-the-shoulder observations of how the work is being done, to assure management of the quality of work necessary to complete the project as required. Data submitted by the operating staff without having the benefits of an auditing program can only be judged as having a confidence level equivalent to the skills of the least trained person performing work on the project. Management must actively pursue a course consistent with the needed confidence level of data.

In spite of records being unavailable, it is felt that the electronic devices used for physical measurements are being maintained sufficiently to provide pressure, level and flow information to a $\pm 2$ percent tolerance of desired nominal values, and temperature information to a $\pm 10$ percent tolerance of desired information (temperature sensors can be calibrated to a $\pm 2$ percent tolerance of a known temperature--the inaccuracies are estimated to be high because of the lack of knowledge of the thermodynamics of the stack gases being measured).

## 5.0 RECOMMENDATIONS

Specifications of a quality control program for the Shawnee scrubber project does not fall within the scope of the project. The recommendations made in the following paragraphs will apply to the implementation of an external (systems review and performance audit) QA program.* This type of program normally should be carried out by an organization which has no special interest in the data; i.e., no self-interest to protect and no preconceptions as to the quality of the information forthcoming. On the other hand, the organization should be reputable and well qualified to carry out the type of auditing program desired.

In the case of EPA demonstration projects, EPA may wish to contract a third party to handle the audit program, or it may handle the program by means of its own QA staff. In either case, it is quite important that the auditing be done competently and objectively.

It is recommended that the wet limestone scrubber operation located at the Shawnee steam-electric plant, Paducah, Kentucky, be externally audited twice each calendar year. Timing of the audit program, which normally should take 1 calendar week, should be coordinated among the auditing team, EPA, TVA, and the Bechtel Corporation. Some advance notice is necessary in order to insure cooperation of operational personnel. It is not recommended that the audits be scheduled on a regular basis, since by definition an audit is conducted without extensive "preparation" at the project being audited. Advance notice to EPA and Bechtel supervisory staff should be at least 2 weeks, so that the audit team can be apprised of special test and analysis schedules which may alter its audit procedure or cause postponement of the audit itself. Advance notice to TVA staff (senior chemist, instrumentation foreman) should be at least 1 week.

It is recommended that, for a facility such as the Shawnee wet limestone scrubber unit, the audit team concentrate its efforts in the following major areas:

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*It is important that the Shawnee project develop its own internal QA program, which might well be along the lines suggested here for the external audit and review.

1. Verification of pH measurements at inlet and outlet, on both TCA and venturi scrubbers. An accurate pH meter brought in by the audit team, with appropriate buffer solutions, should be used. Measurement of pH is critical to efficient process control at this facility.

2. Independent chemical analysis of slurry samples, by several laboratories. A continuing external audit program can aid in establishing acceptance limits and method biases.

3. Verification of particulate mass loading and sulfur dioxide measurement systems. If possible, side-by-side operation of TVA and audit team sampling trains should be carried out, with independent analyses of the collected samples. A wet chemical technique such as total acid titration should be used to check the $SO_2$ analyzer response.

   If a duplicate sampling train could be used by the audit team, then critical measurement parameters should be identified and checked. For stack sampling procedure, this includes (at a minimum):
   a. Sample volume measurement check by means of a calibrated wet test or dry gas meter;*
   b. Pitot tube ($C_p$ factor) check by means of an NBS calibrated pitot tube;
   c. Thermometer and thermocouple checks with a calibrated temperature measurement system;
   d. Stack gas moisture content check by means of an absorbing impinger train.

4. Monitoring of process control instrumentation with appropriate electronic devices (precision resistances, calibrated voltage and current meters with digital readout, signal generators).

It is premature to set acceptance limits based on audit data from the Shawnee scrubber. Several observations can be made, however, as follows:

1. pH measurement as currently being made in the scrubber pots should be accurate within 0.2 pH unit, possibly within 0.1 unit. The in-line systems suffer from bias due to flow rate variance, probe surface modifications and the like. A much more sophisticated (and costly) measurement system, employing in-situ self-cleaning probes, could possibly improve the accuracy and precision of the measurement. It should be kept in mind, however, that because of the nature of the medium being measured there is a "built-in" uncertainty in the pH which no measurement system can overcome.

2. Sulfur dioxide concentrations in the stack gas, as read from the du Pont Photometric Analyzer, are probably reliable to $\pm$20 percent.

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* For suggestions as to techniques available, see "Process Stream Volumetric Flow Measurement and Gas Sample Extraction Methodology," by Brooks & Williams. This manual (TRW Document No. 24916-6028-RU-00) was prepared under EPA Contract No. 68-02-1412, for the Process Measurements Branch of IERL.

3. Control laboratory slurry analysis acceptance limits will vary depending on the particular technique but should be controllable generally to ±20 percent. Particular attention should be devoted to defining and quantitating matrix effects in the X-ray fluorescence method, with appropriate corrections for concentration differences. This appears to be a sizable error source.

4. Process instrumentation and physical measurement techniques do not represent sizable error sources at the Shawnee facility, although recordkeeping by instrumentation personnel has been minimal. Generally, instrumentation is reliable and well maintained.

## APPENDIX A COMPARISON ON ANALYSIS OF LIMESTONE SLURRY

Cooperating laboratories were:

1. TVA Power Service Center Laboratory, Chattanooga, Tennessee - Mr. John Rose, contact
2. TVA Power Service Center Laboratory, Muscle Shoals, Alabama - Dr. Guerry McClellon, contact
3. Research Triangle Institute, Research Triangle Park, North Carolina - Dr. D. E. Wagoner, contact

Results from RTI laboratories are presented in two sections. One set of data was obtained on slurry which was filtered at the Shawnee Laboratory. The second set of data results from analysis of samples filtered in the RTI laboratory.

The first eight matrixes present results for each element: calcium, magnesium, and total sulfur in the solid; and calcium, magnesium, sodium, potassium, and chloride in the liquid. The next five matrixes give results of all analyses for each laboratory, with Shawnee results listed first. The last four matrixes break down the total sulfur and calcium analyses into results by a standard "wet" technique, by X-ray fluorescence using Shawnee standard values, and by X-ray fluorescence using RTI-derived standard values on the Shawnee standard material.

Table 1. Analysis for calcium in slurry solid

| Sample: Ca (as CaO) wt % / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 24.73 | 24.50 | 22.78 | 21.05 | 22.87 |
| RTI (Shawnee filtered) | 22.12 | 19.03 | 19.60 | 18.40 | 19.46 |
| RTI (RTI filtered) | 17.99 | 17.96 | 18.63 | 18.13 | 19.32 |
| Chattanooga (TVA) | 23.45 | 23.24 | 23.02 | 22.46 | 22.46 |
| Muscle Shoals (TVA) | 23.6 | 23.1 | 23.6 | 22.6 | 22.9 |

Table 2. Analysis for magnesium in slurry solid

| Sample: Mg (as MgO) wt % / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 0.30 | 0.29 | 0.28 | 0.27 | 0.29 |
| RTI (Shawnee filtered) | 0.43 | 0.48 | 0.39 | 0.36 | 0.42 |
| RTI (RTI filtered) | 0.61 | 0.52 | 0.34 | 0.39 | 0.35 |
| Chattanooga (TVA) | 0.65 | 0.56 | 0.56 | 0.61 | 0.61 |
| Muscle Shoals (TVA) | 0.25 | 0.25 | 0.24 | 0.25 | 0.24 |

Table 3. Analysis for total sulfur in slurry solid

| Laboratory \ Sample: TS (as $SO_3$) wt % | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 34.78 | 34.16 | 31.22 | 28.17 | 31.64 |
| RTI (Shawnee filtered) | 36.70 | 32.00 | 33.03 | 30.53 | 32.80 |
| RTI (RTI filtered) | 28.60 | 27.00 | 31.13 | 28.78 | 32.03 |
| Chattanooga (TVA) | 30.5 | 30.7 | 30.0 | 29.2 | 29.6 |
| Muscle Shoals (TVA) | 31.3 | 30.9 | 31.4 | 29.7 | 30.4 |

Table 4. Analysis for calcium in slurry filtrate

| Sample: Ca (ppm) / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 1720 | 1710 | 1810 | 2090 | 2315 |
| RTI (Shawnee filtered) | 1775 | 1825 | 1810 | 1708 | 1885 |
| RTI (RTI filtered) | 1700 | 1810 | 1730 | 1720 | 1825 |
| Chattanooga (TVA) | 1756 | 1740 | 1676 | 1596 | 1732 |
| Muscle Shoals (TVA) | 1787 | 1787 | 1716 | 1787 | 1716 |

Table 5. Analysis for magnesium in slurry filtrate

| Sample: Mg (ppm) / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 733 | 699 | 662 | 691 | 698 |
| RTI (Shawnee filtered) | 785 | 945 | 813 | 1000 | 1115 |
| RTI (RTI filtered) | 730 | 805 | 805 | 795 | 770 |
| Chattanooga (TVA) | 768 | 734 | 763 | 780 | 816 |
| Muscle Shoals (TVA) | 724 | 724 | 724 | 724 | 784 |

Table 6. Analysis for sodium in slurry filtrate

| Sample: Na (ppm) / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 71 | 57 | 70 | 73 | 82 |
| RTI (Shawnee filtered) | 71 | 69 | 79 | 75 | 77 |
| RTI (RTI filtered) | 153 | 161 | 180 | 176 | 145 |
| Chattanooga (TVA) | 66 | 62 | 64 | 66 | 69 |
| Muscle Shoals (TVA) | 41 | 37 | 37 | 41 | 41 |

Table 7. Analysis for potassium in slurry filtrate

| Sample: K / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 118 | 121 | 123 | 126 | 153 |
| RTI (Shawnee filtered) | 103 | 101 | 105 | 108 | 111 |
| RTI (RTI filtered) | 116 | 102 | 114 | 118 | 116 |
| Chattanooga (TVA) | 107 | 96 | 107 | 116 | 116 |
| Muscle Shoals (TVA) | 58 | 54 | 50 | 58 | 58 |

Table 8. Analysis for chloride in slurry filtrate

| Sample: Cl (ppm) / Laboratory | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Shawnee | 3651 | 3580 | 3545 | 3545 | 3580 |
| RTI (Shawnee filtered) | 3697 | 3700 | 3855 | 3660 | 3987 |
| RTI (RTI filtered) | 3621 | 3638 | 3754 | 3519 | 3566 |
| Chattanooga (TVA) | 3692 | 3543 | 3571 | 3571 | 3628 |
| Muscle Shoals (TVA) | 3800 | 3700 | 3600 | 3600 | 3700 |

Table 9. Laboratory: Shawnee (TVA)

| SOLID (Wt. %) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Ca (CaO) | 24.73 | 19.03 | 22.78 | 21.05 | 22.87 |
| Mg (MgO) | 0.30 | 0.48 | 0.28 | 0.27 | .29 |
| TS ($SO_3$) | 34.78 | 32.00 | 31.22 | 28.17 | 31.64 |
| LIQUID (ppm) | | | | | |
| Ca | 1720 | 1710 | 1810 | 2090 | 2315 |
| Mg | 733 | 699 | 662 | 691 | 698 |
| Na | 71 | 57 | 70 | 73 | 82 |
| K | 118 | 121 | 123 | 126 | 153 |
| Cl | 3651 | 3580 | 3545 | 3545 | 3580 |

Table 10. Laboratory: RTI (Shawnee filtered)

| SOLID (Wt. %) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Ca (CaO) | 22.12 | 19.03 | 19.10 | 18.41 | 19.46 |
| Mg (MgO) | 0.43 | 0.48 | 0.39 | 0.36 | 0.42 |
| TS ($SO_3$) | 36.7 | 32.00 | 33.03 | 30.53 | 32.80 |
| LIQUID (ppm) | | | | | |
| Ca | 1775 | 1825 | 1810 | 1708 | 1885 |
| Mg | 785 | 945 | 913 | 1000 | 1115 |
| Na | 71 | 69 | 79 | 75 | 77 |
| K | 103 | 101 | 105 | 108 | 111 |
| | | | | | |
| Cl | 3697 | 3700 | 3855 | 3660 | 3987 |

Table 11. Laboratory: RTI

| SOLID (Wt. %) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Ca (CaO) | 17.99 | 19.96 | 18.63 | 18.13 | 19.32 |
| Mg (MgO) | 0.61 | 0.52 | 0.34 | 0.39 | 0.35 |
| TS ($SO_3$) | 28.6 | 27.0 | 31.13 | 28.78 | 32.03 |
| LIQUID (ppm) | | | | | |
| Ca | 1700 | 1810 | 1730 | 1720 | 1825 |
| Mg | 730 | 805 | 805 | 795 | 770 |
| Na | 153 | 101 | 180 | 176 | 145 |
| K | 116 | 102 | 114 | 118 | 116 |
| | | | | | |
| Cl | 3621 | 3638 | 3754 | 3519 | 3566 |

Table 12. Laboratory: Muscle shoals (TVA)

| SOLID (Wt. %) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Ca (CaO) | 23.6 | 23.1 | 23.6 | 22.6 | 22.9 |
| Mg (MgO) | 0.25 | 0.25 | 0.24 | 0.25 | 0.24 |
| TS ($SO_3$) | 31.3 | 30.9 | 31.4 | 29.7 | 36.4 |
| LIQUID (ppm) | | | | | |
| Ca | 1787 | 1787 | 1716 | 1787 | 1716 |
| Mg | 724 | 724 | 724 | 724 | 784 |
| Na | 41 | 37 | 37 | 41 | 41 |
| K | 58 | 54 | 50 | 58 | 58 |
| | | | | | |
| Cl | 3800 | 3700 | 3600 | 3600 | 3700 |

Table 13. Laboratory: Chattanooga (TVA)

| SOLID (Wt. %) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| Ca (CaO) | 23.45 | 23.24 | 23.02 | 22.46 | 22.46 |
| Mg (MgO) | 0.65 | 0.56 | 0.56 | 0.61 | 0.61 |
| TS ($SO_3$) | 30.5 | 30.7 | 30.0 | 29.2 | 29.6 |
| LIQUID (ppm) | | | | | |
| Ca | 1756 | 1740 | 1676 | 1596 | 1732 |
| Mg | 768 | 734 | 763 | 780 | 816 |
| Na | 66 | 62 | 64 | 66 | 67 |
| K | 107 | 96 | 107 | 116 | 116 |
| | | | | | |
| Cl | 3692 | 3543 | 3571 | 3571 | 3628 |

Table 14. Total sulfur determinations, Shawnee filtered samples analyzed at RTI

| wt % (as $SO_3$) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| By $BaCl_2$ precipitation | 36.7 | 32.00 | 33.03 | 30.53 | 32.80 |
| By X-Ray fluroescence | | | | | |
| Using Shawnee X-Ray standard number for TS | 22.08* | 21.25 | 23.15 | 27.75 | 22.95 |
| Using RTI determined number for TS | | | | | |
| Shawnee X-Ray Standard(as $SO_3$)* | | | | | |
| Shawnee given | 28.45 | | | | |
| RTI determined | 28.38 | | | | |

*Shawnee and RTI TS determinations on the XRF standard were virtually idential, so the Shawnee TS value only was used in calculating wt % TS in each sample.

Table 15. Total sulfur determinations, RTI filtered and analyzed samples

| | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| $BaCl_2$ precipitation | 28.6 | 27.00 | 31.13 | 28.78 | 32.03 |
| X-Ray fluorescence | | | | | |
| Using Shawnee X-Ray standard number for TS | 35.13 | 30.28 | 29.8 | 34.45 | 29.18 |
| Using RTI-determined number for TS | | | | | |

Table 16. Calcium determinations, Shawnee filtered samples analyzed at RTI

| wt % (as CaO) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| By AA | 22.12 | 19.03 | 19.60 | 18.41 | 19.46 |
| By X-Ray fluorescence | | | | | |
| Using Shawnee X-Ray standard number for CaO | 23.13 | 25.41 | 22.25 | 25.76 | 22.89 |
| Using RTI determined number for CaO | 20.09 | 22.06 | 19.32 | 22.37 | 19.88 |
| Shawnee X-Ray Standard (as CaO) | | | | | |
| Shawnee given | 25.41 | | | | |
| RTI determined | 22.06 | | | | |

Table 17. Calcium determinations, RTI filtered and analyzed samples

| wt % (as CaO) | 11/18/75 | | | 11/19/75 | |
|---|---|---|---|---|---|
| | 1100 | 1500 | 2300 | 1100 | 2300 |
| By AA | 17.99 | 17.96 | 18.63 | 18.13 | 19.32 |
| By X-Ray fluorescence | | | | | |
| Using Shawnee X-Ray standard number for CaO | 28.35 | 25.52 | 26.17 | 27.68 | 26.32 |
| Using RTI-determined number for CaO | 24.61 | 22.16 | 22.72 | 24.04 | 22.85 |

## APPENDIX B QUALITATIVE SYSTEMS REVIEW FOR THE CONTROL LABORATORY

This checklist is designed to:

1. Identify existing system deocumentation; i.e., maintenance manuals, organizational structure, operating procedures, etc.
2. Evaluate the adequacy of the procedures as documented.
3. Evaluate the degree of use of and adherence to the documented procedures in day-to-day operations based on observed conditions (auditor) and a review of applicable records on file.

The checklist gives three descriptions to each facet of a quality control system. In all cases the "5" choice is the most desirable and effective mode of operation; "3" is marginal and tolerable; "1" is definitely unacceptable and ineffective as a mode of operation.

It is not always possible to describe accurately all options with only three choices. Therefore, a "2" or "4" rating may be selected if the evaluator feels that an in-between score is more descriptive of the actual situation.

After all the applicable questions are answered, an average is computed to give an overall indication of the quality system effectiveness.

Generally, a rating of 3.8 or better is considered acceptable.

A rating between 2.5 and 3.8 indicates a need for improvement but there is no imminent threat to project performance as it now stands.

For the control laboratory, the results are as follows:

a. Of 82 check questions, 65 were answered on site;
b. Average score was 3.0 (5.0 maximum), indicating a satisfactory but not outstanding program as presently operated;
c. The control laboratory was judged weak in its quality control organization, procurement and inventory procedures, and personnel training policy;
d. Strong points were its day-to-day "in-process" quality assurance, its calibration procedures, and its facilities and equipment.

The completed questionnaire, with indicated judgments in specific areas, is given herewith. These judgments are for the control laboratory operation only.

QUALITY ORGANIZATION

| | | SCORE |
|---|---|---|
| (1) | Overall responsibility for quality assurance (or quality control) for the organization is: | |
| | (a) Assigned to one individual by title (e.g., Quality Control Coordinator). | 5 |
| | (b) Assigned to a specific group within the organization. | 3 |
| ✓ | (c) Not specifically assigned but left to the discretion of the various operation, analytical, inspection, and testing personnel. | 1 |
| (2) | The Quality Control Coordinator is located in the organization such that: | |
| | (a) He has direct access to the top management level for the total operation independent of others involved in operational activities. | 5 |
| | (b) He performs as a peer with others involved in operational activities with access to top management through the normal chain of command. | 3 |
| ✓ | (c) His primary resonsibility is in operational activities with quality assurance as an extra or part-time effort. | 1 |
| (3) | Data reports are distributed to: | |
| ✓ | (a) All levels of management.* | 5 |
| | (b) One level of management only. | 3 |
| | (c) The quality control group only. | 1 |
| (4) | Data Quality Reports contain: | |
| | (a) Information of operation trends, required actions, and danger spots. | 5 |
| ✓ | (b) Information on suspected data/analyses and their causes | 3 |
| | (c) Percent of valid data per month. | 1 |

*Management appropriate levels in all applicable organizations such as subcontractors, prime contractor, EPA.

SCORE

(5) The quality control system is:

(a) Formalized and documented by a set of procedures which clearly describe the activities necessary and sufficient to achieve desired quality objectives from procurement through to reporting data to the EPA/RTP. 5

(b) Contained in methods procedures or is implicit in those procedures. Experience with the materials, product, and equipment is needed for continuity of control. 3

✓ (c) Undefined in any procedures and is left to the current managers or supervisors to determine as the situation dictates. 1

(6) Support for quality goals and results is indicated by:

(a) A clear statement of quality objectives by the top executive with continuing visible evidence of its sincerity to all levels of the organization. 5

✓ (b) Periodic meetings among operations personnel and the individual(s) responsible for quality assurance on quality objectives and progress toward their achievement. 3

(c) A "one-shot" statement of the desire for product quality by the top executive after which the quality assurance staff is on its own. 1

(7) Accountability for quality is:

(a) Clearly defined for all sections and operators/analysts where their actions have an impact on quality. 5

(b) Vested with the Quality Control Coordinator who must use whatever means possible to achieve quality goals. 3

✓ (c) Not defined. 1

THE QUALITY SYSTEM (continued)

SCORE

(8) The acceptance criteria for the level of quality of the demonstration projects routine performance are:

(a) Clearly defined in writing for all characteristics. 5

(b) Defined in writing for some characteristics and some are dependent on experience, memory, and/or verbal communication. 3

✓ (c) Only defined by experience and verbal communication. 1

(9) Acceptance criteria for the level of quality of the project's routine performance are determined by:

(a) Monitoring the performance in a structured program of inter- and intralaboratory evaluations. 5

✓ (b) Scientific determination of what is technically feasible. 3

(c) Laboratory determination of what can be done using currently available equipment, techniques, and manpower. 1

(10) Decisions on acceptability of questionable results are made by:

✓ (a) A review group consisting of the chief chemist or engineer, quality control, and others who can render expert judgment. 5

(b) An informal assessment by quality control. 3

(c) The operator/chemist. 1

NA (11) The quality control coordinator has the authority to:

(a) Affect the quality of analytical results by inserting controls to assure that the methods meet the requirements for precision, accuracy, sensitivity, and specificity. 5

(b) Reject suspected results and stop any method that produces high levels of discrepancies. 3

(c) Submit suspected results to management for a decision on disposition. 1

IN-PROCESS QUALITY ASSURANCE

SCORE

(12) Measurement methods are checked:

✓ (a) During operation for conformance to operating conditions and to specifications; e.g., flow rates, reasonableness of data, etc. 5

(b) During calibration to determine acceptability of the results. 3

(c) Only when malfunctions are reported. 1

(13) The capability of the method to produce within specification limit is:

✓ (a) Known through method capability analysis ($\bar{X}$-R Charts) to be able to produce consistently acceptable results. 5

(b) Assumed to be able to produce a reasonably acceptable result. 3

(c) Unknown. 1

(14) Method determination discrepancies are:

(a) Analyzed immediately to seek out the cause and apply corrective action. 5

✓ (b) Checked out when time permits. 3

(c) Not detectable with present controls and procedures. 1

(15) The operating conditions (e.g., flow rate, range, temperature, etc.) of the methods are:

✓ (a) Clearly defined in writing in the method for each significant variable. 5

(b) Controlled by supervision based on general guidelines. 3

(c) Left up to the operator/analyst. 1

## IN-PROCESS QUALITY ASSURANCE (continued)

| | SCORE |
|---|---|
| (16) Auxiliary measuring, gaging, and analytical instruments are: | |
| (a) Maintained operative, accurate, and precise by regular checks and calibrations against stable standards which are traceable to the U.S. Bureau of Standards. | 5 |
| (b) Periodically checked against a zero point or other reference and examined for evidence of physical damage, wear, or inadequate maintenance. | 3 |
| (c) Checked only when they stop working or when excessive defects are experienced which can be traced to inadequate instrumentation. | 1 |

## CONFIGURATION CONTROL

SCORE

NA (17) Procedures for documenting, for the record, any design change in the system are:

(a) Written down and readily accessible to those individuals responsible for configuration control. 5

(b) Written down but not in detail. 3

(c) Not documented. 1

(18) Engineering schematics are:

(a) Maintained current on the system and subsystem levels. 5

(b) Maintained current on certain subsystems only. 3

(c) Not maintained current. 1

(19) All computer programs are:

(a) Documented and flow charted. 5

(b) Flow charted. 3

(c) Summarized. 1

? (20) Procedures for transmitting significant design changes in hardware and/or software to the EPA project officer are:

(a) Documented in detail sufficient for implementation. 5

(b) Documented too briefly for implementation. 3

(c) Not documented. 1

## DOCUMENTATION CONTROL

SCORE

(21) Procedures for making revisions to technical documents are:

(a) Clearly spelled out in written form with the line of authority indicated and available to all involved personnel. 5

✓ (b) Recorded but not readily available to all personnel. 3

(c) Left to the discretion of present supervisors/ managers. 1

(22) In revising technical documents, the revisions are:

(a) Clearly spelled out in written form and distributed to all parties affected on a controlled basis which assures that the change will be implemented and permanent. 5

✓ (b) Communicated through memoranda to key people who are responsible for effecting the change through whatever method they choose. 3

(c) Communicated verbally to operating personnel who then depend on experience to maintain continuity of the change. 1

(23) Changes to technical documents pertaining to operational activities are:

(a) Analyzed to make sure that any harmful side effects are known and controlled prior to revision effectivity. 5

✓ (b) Installed on a trial or gradual basis, monitoring the product to see if the revision has a net beneficial effect. 3

(c) Installed immediately with action for correcting side effects taken if they show up in the final results. 1

## DOCUMENTATION CONTROL (continued)

SCORE

(24) Revisions to technical documents are:

✓ (a) Recorded as to date, serial number, etc. when the revision becomes effective. 5

(b) Recorded as to the date the revision was made on written specifications. 3

(c) Not recorded with any degree of precision. 1

NA (25) Procedures for making revisions to computer software programs are:

(a) Clearly spelled out in written form with the line of authority indicated. 5

(b) Not recorded but changes must be approved by the present supervisor/manager. 3

(c) Not recorded and left to the discretion of the programmer. 1

NA (26) In revising software program documentation, the revisions are:

(a) Clearly spelled out in written form with reasons for the change and the authority for making the change distributed to all parties affected by the change. 5

(b) Incorporated by the programmer and communicated through memoranda to key people. 3

(c) Incorporated by the programmer at his will. 1

DOCUMENTATION CONTROL (continued)

| | | | SCORE |
|---|---|---|---|
| NA | (27) | Changes to software program documentation are: | |
| | | (a) Analyzed to make sure that any harmful side effects are known and controlled prior to revision effectivity. | 5 |
| | | (b) Incorporated on a trial basis, monitoring the results to see if the revision has a net beneficial effect. | 3 |
| | | (c) Incorporated immediately with action for detecting and correcting side effects taken as necessary. | 1 |
| NA | (28) | Revisions to software program documentation are: | |
| | | (a) Recorded as to date, program name or number, etc., when the revision becomes effective. | 5 |
| | | (b) Recorded as to the date the revision was made. | 3 |
| | | (c) Not recorded with any degree of precision. | 1 |

## PREVENTIVE MAINTENANCE

| | | | SCORE |
|---|---|---|---|
| (29) | Preventative maintenance procedures are: | | |
| | (a) | Clearly defined and written for all measurement systems and support equipment. | 5 |
| ✓ | (b) | Clearly defined and written for most of the measurement systems and support equipment. | 3 |
| | (c) | Defined and written for only a small fraction of the total number of systems. | 1 |
| (30) | Preventative maintenance activities are documented: | | |
| | (a) | On standard forms in station log books. | 5 |
| ✓ | (b) | Operator/analyst summary in log book. | 3 |
| | (c) | As operator/analyst notes. | 1 |
| (31) | Preventative maintenance procedures as written appear adequate to insure proper equipment operation for: | | |
| | (a) | All measurement systems and support equipment. | 5 |
| ✓ | (b) | Most of the measurement systems and support equipment. | 3 |
| | (c) | Less than half of the measurement systems and support equipment. | 1 |
| (32) | A review of the preventative maintenance records indicates that: | | |
| ✓ | (a) | Preventative maintenance procedures have been carried out on schedule and completely documented. | 5 |
| | (b) | The procedures were carried out on schedule but not completely documented. | 3 |
| | (c) | The procedures were not carried out on schedule all the time and not always documented. | 1 |

PREVENTATIVE MAINTENANCE (continued)

SCORE

? (33) Preventative maintenance records (histories) are:

(a) Utilized in revising maintenance schedules, developing an optimum parts/reagents inventory and development of scheduled replacements to minimize wear-out failures. 5

(b) Utilized when specific questions arise and for estimating future work loads. 3

(c) Utilized only when unusual problems occur. 1

## DATA VALIDATION PROCEDURES

SCORE

(34) Data validation procedures are:

(a) Clearly defined in writing for all measurement systems. 5

(b) Defined in writing for some measurement systems, some dependent on experience, memory, and/or verbal communication. 3

✓ (c) Only defined by experience and verbal communication. 1

(35) Data validation procedures are:

✓ (a) A coordinated combination of computerized and manual checks applied at different levels in the measurement process. 5

(b) Applied with a degree of completeness at no more than two levels of the measurement process. 3

(c) Applied at only one level of the measurement process. 1

(36) Data validation criteria are documented and include:

(a) Limits on: (1) operational parameters such as as flow rates; (2) calibration data; (3) special checks unique to each measurement; e.g., successive values/averages; (4) statistical tests; e.g., outliers; (5) manual checks such as hand calculations. 5

(b) Limits on the above type checks for most of the measurement systems. 3

✓ (c) Limits on some of the above type checks for only the high priority measurements. 1

## DATA VALIDATION PROCEDURES (continued)

| | | | SCORE |
|---|---|---|---|
| | (37) | Acceptable limits as set are reasonable and adequate to insure the detection of invalid data with a high probability for: | |
| | | (a) All measurement systems. | 5 |
| | | (b) At least 3/4 of the measurement systems. | 3 |
| ✓ | | (c) No more than 1/2 of the measurement systems. | 1 |
| | (38) | Data validation activities are: | |
| | | (a) Recorded on standard forms at all levels of the measurement process. | 5 |
| ✓ | | (b) Recorded in the operator's/analyst log book. | 3 |
| | | (c) Not recorded in any prescribed manner. | 1 |
| | (39) | Examination of data validation records indicates that: | |
| ✓ | | (a) Data validation activities have been carried out as specified and completely documented. | 5 |
| | | (b) Data validation activities appear to have been performed but not completely documented. | 3 |
| | | (c) Data validation activities, if performed, are not formally documented. | 1 |
| | (40) | Data validation summaries are: | |
| ✓ | | (a) Prepared at each level or critical point in the measurement process and forwarded to the next level with the applicable block of data. | 5 |
| | | (b) Prepared by and retained at each level. | 3 |
| | | (c) Not prepared at each level nor communicated between levels. | 1 |

DATA VALIDATION PROCEDURES (continued)

SCORE

(41) Procedures for deleting invalidated data are:

(a) Clearly defined in writing for all levels of the measurement process, and invalid data are automatically deleted when one of the computerized validation criteria are exceeded. 5

(b) Programmed for automatic deletion when computerized validation criteria are exceeded but procedures not defined when manual checks detect invalid data. 3

✓ (c) Not defined for all levels of the measurement process. 1

(42) Quality audits (i.e., both on-site system reviews and/or quantitative performance audits) independent of the normal operations are:

(a) Performed on a random but regular basis to insure and quantify data quality. 5

✓ (b) Performed whenever a suspicion arises that there are areas of ineffective performance. 3

(c) Never performed. 1

## PROCUREMENT AND INVENTORY PROCEDURES

SCORE

(43) Purchasing guidelines are established and documented for:

(a) All equipment and reagents having an effect on data quality. 5

(b) Major items of equipment and critical reagents. 3

✓ (c) A very few items of equipment and reagents. 1

(44) Performance specifications are:

(a) Documented for all items of equipment which have
✓ an effect on data quality. 5

(b) Documented for the most critical items only. 3

(c) Taken from the presently used items of equipment. 1

(45) Reagents and chemicals (critical items) are:

(a) Procured from suppliers who must submit samples for test and approval prior to initial shipment. 5

(b) Procured from suppliers who certify they can meet
✓ all applicable specifications. 3

(c) Procured from suppliers on the basis of price and delivery only. 1

(46) Acceptance testing for incoming equipment is:

(a) An established and documented inspection procedure to determine if procurements meet the quality assurance and acceptance requirements. Results are documented. 5

(b) A series of undocumented performance tests performed by the operator before using the equipment. 3

✓ (c) The receiving document is signed by the responsible individual indicating either acceptance or rejection. 1

## PROCUREMENT AND INVENTORY PROCEDURES

| | | | SCORE |
|---|---|---|---|
| | (47) | Reagents and chemicals are: | |
| | | (a) Checked 100 percent against specification, quantity, and for certification where required and accepted only if they conform to all specifications. | 5 |
| ✓ | | (b) Spot-checked for proper quantity and for shipping damage. | 3 |
| | | (c) Released to analyst by the receiving clerk without being checked as above. | 1 |
| | (48) | Information on discrepant purchased materials is: | |
| | | (a) Transmitted to the supplier with a request for corrective action. | 5 |
| ✓ | | (b) Filed for future use. | 3 |
| | | (c) Not maintained. | 1 |
| ? | (49) | Discrepant purchased materials are: | |
| | | (a) Submitted to a review by Quality Control and Chief Chemist for disposition. | 5 |
| | | (b) Submitted to Service Section for determination on acceptability. | 3 |
| | | (c) Used because of scheduling requirements. | 1 |
| | (50) | Inventories are maintained on: | |
| | | (a) First-in, first-out basis. | 5 |
| ✓ | | (b) Random selection in stock room. | 3 |
| | | (c) Last-in, first-out basis. | 1 |

PROCUREMENT AND INVENTORY PROCEDURES (continued)

| | | | SCORE |
|---|---|---|---|
| (51) | Receiving of materials is: | | |
| | (a) | Documented in a receiving record log giving a description of the material, the date of receipt, results of acceptance test, and the signature of the responsible individual. | 5 |
| | (b) | Documented in a receiving record log with material title, receipt date, and initials of the individual logging the material in. | 3 |
| ✓ | (c) | Documented by filing a signed copy of the requisition. | 1 |
| (52) | Inventories are: | | |
| | (a) | Identified as to type, age, and acceptance status. | 5 |
| | (b) | Identified as to material only. | 3 |
| ✓ | (c) | Not identified in writing. | 1 |
| (53) | Reagents and chemicals which have limited shelf life are: | | |
| | (a) | Identified as to shelf life expiration date and systematically issued from stock only if they are still within that date. | 5 |
| | (b) | Issued on a first-in, first-out basis, expecting that there is enough safety factor so that the expiration date is rarely exceeded. | 3 |
| ✓ | (c) | Issued at random from stock. | 1 |

## PERSONNEL TRAINING PROCEDURES

SCORE

(54) Training of new employees is accomplished by:

(a) A programmed system of training where elements of training, including quality standards, are included in a training checklist. The employee's work is immediately rechecked by supervisors for errors or defects and the information is fed back instantaneously for corrective action. 5

(b) On-the-job training by the supervisor who gives an overview of quality standards. Details of quality standards are learned as normal results are fed back to the chemist. 3

✓ (c) On-the-job learning with training on the rudiments of the job by senior coworkers. 1

(55) When key personnel changes occur:

(a) Specialized knowledge and skills are retained in the form of documented methods and descriptions. 5

(b) Replacement people can acquire the knowledge of their predecessors from coworkers, supervisors, and detailed study of the specifications and memoranda. 3

✓ (c) Knowledge is lost and must be regained through long experience or trial-and-error. 1

(56) The people who have an impact on quality; e.g., calibration personnel, maintenance personnel, bench chemists, supervisors, etc., are:

(a) Trained in the reasons for and the benefits of standards of quality and the methods by which high quality can be achieved. 5

(b) Told about quality only when their work falls below acceptable levels. 3

✓ (c) Are reprimanded when quality deficiencies are directly traceable to their work. 1

PERSONNEL TRAINING PROCEDURES (continued)

SCORE

(57) The employee's history of training accomplishments is maintained through:

(a) A written record maintained and periodically reviewed by the supervisor. 5

(b) A written record maintained by the employee. 3

✓ (c) The memory of the supervisor/employee. 1

(58) Employee proficiency is evaluated on a continuing basis by:

(a) Periodic testing in some planned manner with the results of such tests recorded. 5

(b) Testing when felt necessary by the supervisor. 3

✓ (c) Observation of performance by the supervisor. 1

NA (59) Results of employee proficiency tests are:

(a) Used by management to establish the need for and type of special training. 5

(b) Used by the employee for self-evaluation of needs. 3

(c) Used mostly during salary reviews. 1

## FEEDBACK AND CORRECTIVE ACTION

SCORE

(60) A feedback and corrective action mechanism to assure that problems are reported to those who can correct them and that a closed loop mechanism is established to assure that appropriate corrective actions have been taken is:

(a) Clearly defined in writing with individuals assigned specific areas of responsibility. 5

(b) Written in general terms with no assignment of responsibilities. 3

✓ (c) Not formalized but left to the present supervisors/managers. 1

(61) Feedback and corrective action activities are:

(a) Documented on standard forms. 5

✓ (b) Documented in the station log book. 3

(c) Documented in the operator's/analyst's notebook. 1

? (62) A review of corrective action records indicates that:

(a) Corrective actions were systematic, timely, and fully documented. 5

(b) Corrective actions were not always systematic, timely, or fully documented. 3

(c) A closed loop mechanism did not exist. 1

? (63) Periodic summary reports on the status of corrective action are distributed by the responsible individual to:

(a) All levels of management. 5

(b) One level of management only. 3

(c) The group generating the report only. 1

FEEDBACK AND CORRECTIVE ACTION (continued)

| | | | SCORE |
|---|---|---|---|
| (64) | The reports include: | | |
| | (a) | A listing of major problems for the reporting period; names of persons responsible for corrective actions; criticality of problems; due dates; present status; trend of quality performance (i.e., response time, etc.); listing of items still open from previous reports. | 5 |
| | (b) | Most of the above items. | 3 |
| ✓ | (c) | Present status of problems and corrective actions. | 1 |

## CALIBRATION PROCEDURES

SCORE

(65) Calibration procedures are:

✓ (a) Clearly defined and written out in step-by-step fashion for each measurement system and support device. 5

(b) Defined and summarized for each system and device. 3

(c) Defined but operational procedures developed by the individual. 1

(66) Calibration procedures as written are:

✓ (a) Judged to be technically sound and consistent with data quality requirements. 5

(b) Technically sound but lacking in detail. 3

(c) Technically questionable and lacking in detail. 1

(67) Calibration standards are:

(a) Specified for all systems and measurement devices with written procedures for assuring, on a continuing basis, traceability to primary standards. 5

✓ (b) Specified for all major systems with written procedures for assuring traceability to primary standards. 3

(c) Specified for all major systems but no procedures for assuring traceability to primary standards. 1

(68) Calibration standards and traceability procedures as specified and written are:

✓ (a) Judged to be technically sound and consistent with data quality requirements. 5

(b) Standards are satisfactory but traceability is not verified frequently enough. 3

(c) Standards are questionable. 1

CALIBRATION PROCEDURES (continued)

| | | | SCORE |
|---|---|---|---|
| | (69) | Frequency of calibration is: | |
| | | (a) Established and documented for each measurement system and support measurement device | 5 |
| ✓ | | (b) Established and documented for each major measurement system. | 3 |
| | | (c) Established and documented for each air quality measurement system. | 1 |
| | (70) | A review of calibration data indicates that the frequency of calibration as implemented: | |
| | | (a) Is adequate and consistent with data quality requirements. | 5 |
| ✓ | | (b) Results in limits being exceeded a small fraction of the time. | 3 |
| | | (c) Results in limits being exceeded frequently. | 1 |
| | (71) | A review of calibration history indicates that: | |
| | | (a) Calibration schedules are adhered to and results fully documented. | 5 |
| ✓ | | (b) Schedules are adhered to most of the time. | 3 |
| | | (c) Schedules are frequently not adhered to. | 1 |
| ? | (72) | A review of calibration history and data validation records indicates that: | |
| | | (a) Data are always invalidated and deleted when calibration criteria are exceeded. | 5 |
| | | (b) Data are not always invalidate and/or deleted when criteria are exceeded. | 3 |
| | | (c) Data are frequently not invalidated and/or deleted when criteria are exceeded. | 1 |

CALIBRATION PROCEDURES (continued)

SCORE

(73) Acceptability requirements for calibration results are:

(a) Defined for each system and/or device requiring calibration including elapsed time since the last calibration as well as maximum allowable change from the previous calibration. 5

✓ (b) Defined for all major measurement systems. 3

(c) Defined for some major measurements systems only. 1

(74) Acceptability requirements for calibration results as written are:

(a) Adequate and consistent with data quality requirements. 5

✓ (b) Adequate but others should be added. 3

(c) Inadequate to insure data of acceptable quality. 1

? (75) Calibration records (histories) are:

(a) Utilized in revising calibration schedules (i.e., frequency). 5

(b) Utilized when specific questions arise and reviewed periodically for trends, completeness, etc. 3

(c) Utilized only when unusual problems occur. 1

## FACILITIES/EQUIPMENT

| | | | SCORE |
|---|---|---|---|
| | (76) | Facilities/Equipment are: | |
| ✓ | (a) | Adequate to obtain acceptable results. | 5 |
| | (b) | Adequate to obtain acceptable results most of the time. | 3 |
| | (c) | Additional facilities and space are needed. | 1 |
| | (77) | Facilities, equipment, and materials are: | |
| ✓ | (a) | As specified in appropriate documentation and/or standards. | 5 |
| | (b) | Generally as specified in appropriate standards. | 3 |
| | (c) | Frequently different from specifications. | 1 |
| | (78) | Housekeeping reflects an orderly, neat, and effective attitude of attention to detail in: | |
| ✓ | (a) | All of the facilities. | 5 |
| | (b) | Most of the facilities. | 3 |
| | (c) | Some of the facilities. | 1 |
| ? | (79) | Maintenance Manuals are: | |
| | (a) | Complete and readily accessible to maintenance personnel for all systems, components, and devices. | 5 |
| | (b) | Complete and readily accessible to maintenance personnel for all major systems, components, and devices. | 3 |
| | (c) | Complete and accessible for only a few of the systems. | 1 |

## RELIABILITY

SCORE

(80) Procedures for reliability data collection, processing, and reporting are:

(a) Clearly defined and written for all system components. 5

✓ (b) Clearly defined and written for major components of the system. 3

(c) Not defined. 1

(81) Reliability data are:

(a) Recorded on standard forms. 5

✓ (b) Recorded as operator/analyst notes. 3

(c) Not recorded. 1

(82) Reliability data are:

✓ (a) Utilized in revising maintenance and/or replacement schedules. 5

(b) Utilized to determine optimum parts inventory. 3

(c) Not utilized in any organized fashion. 1

## APPENDIX C RTI SULFUR DIOXIDE ANALYTICAL PROCEDURES

### Total Aid Determination

#### Sampling

Samples were obtained by absorbing $SO_2$ from the stack gas in an impinger train containing 3% $H_2O_2$. A sample volume of approximately 10 $\ell$ of sample was pulled through the impingers per run. The contents of the impingers as well as a rinse of 3% $H_2O_2$ were transferred to a labelled sample bottle and shipped to the RTI laboratory for analysis. In addition an appropriate blank was prepared for all absorbing reagents.

#### Analysis

Each sample was diluted to 100 m$\ell$ and a 15-m$\ell$ aliquot was titrated to a bromophenol blue endpoint with 0.01 N sodium hydroxide. A pH meter and probe were used to detect the final endpoint. Reproducibility of the titrations was 0.1 percent or less.

### Determination by Barium Chloranilate

#### Sampling

Samples were obtained from the stack gas by a sampling train containing 80% IPN* in the first impinger (bubbler) and 3% $H_2O_2$ in the second and third impingers. The sample volume was approximately 10 $\ell$. All samples, rinses, and appropriate blanks were shipped to the RTI laboratory for analysis.

#### Analysis

The 80% IPN samples were titrated as a total acid sample. The 3% $H_2O_2$ samples were diluted to 100 m$\ell$ and a 10-m$\ell$ aliquot was taken for analysis. The pH of the aliquot was adjusted to slightly acidic with 1N HCl and buffered with 5 m$\ell$ of 5.6 buffer. The sample then was diluted with alcohol and solid barium chloranilate added. The sample was shaken for 20 minutes and centrifuged at 2,800 rpm. The sample absorbance was determined in a 1-cm cell at a wavelength of 530 nm.

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*Isopropanol.